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X21

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Real-Time X-Ray Studies of Gallium Adsorption and Desorption

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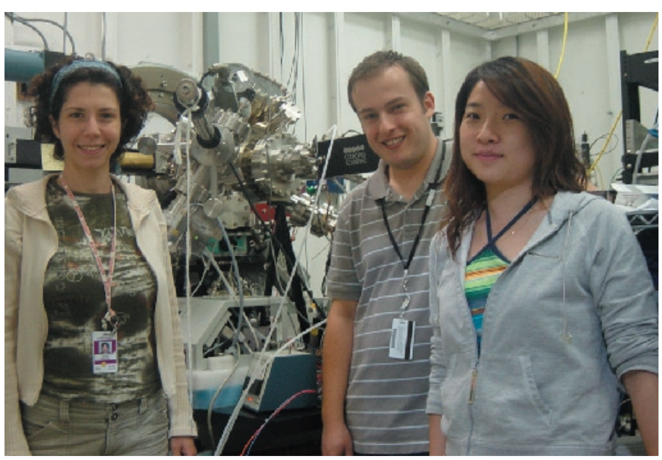
Real-time grazing-incidence small-angle x-ray scattering has been employed to study the adsorption and desorption of Ga on c-plane sapphire and Ga-polar GaN surfaces. Formation of liquid Ga nano-droplets has been observed on sapphire during Ga exposure from an effusion cell at high flux. Following the Ga deposition, the nano-droplets were nitridated in situ by a nitrogen plasma source, which converted the droplets into GaN nanodots. In addition to the droplet studies, at lower Ga flux the adsorption and desorption of Ga has been studied in the pre-droplet regime. Significantly different Ga adsorption/desorption rates were observed on sapphire and GaN surfaces.

Wide-bandgap III-V nitrides (e.g. GaN) have important applications in optoelectronics and high-power devices. During the growth of these materials by molecular beam epitaxy (MBE) the best films are grown in a Ga-rich environment; it is believed that the excess Ga forms a wetting layer on the surface that promotes lateral diffusion of atoms arriving at the growing surface. Thus, it is important to understand the kinetics and behavior of Ga adsorption and desorption on sapphire substrates and GaN films. We have recently used the NSLS for the real-time study of surface- and thin-film processes on beamline X21 to investigate these issues.

Figure 1 shows the real-time grazing-incidence small-angle x-ray scattering (GISAXS) evolution during Ga adsorption onto c-plane sapphire at 700 °C. Initially, the scattering profile shows only the intensity of the near-specular reflection from the sample surface. At approximately t = 15 s, the Ga shutter was opened and, as Ga exposure continues, the broad diffuse scattering background evolves into symmetric peaks centered on approximately $|q_{//}| = 0.12\ nm^{-1}$. These peaks indicate the formation of laterally correlated Ga droplets with an average real-space separation of approximately 50 nm.

Absorption and desorption rates on sapphire substrates were examined systematically as a function of Ga deposition rate and substrate temperature by monitoring the Ga K-edge fluorescence, which is proportional to the number of Ga atoms on the surface, as shown in **Figure 2**. These are being analyzed to determine the activation energy for Ga desorption and the temperature dependence of the Ga sticking coefficient.

After formation of Ga nanodroplets on the sapphire surface, some of the samples were nitridated with a nitrogen plasma to form GaN nanodots. This allowed the real-space morphology of the droplets to be examined by post-facto atomic force microscopy (AFM) and electron microscopy with results that can be compared to existing simulations of droplet nucleation, growth, and coalescence. In addition, we found that this nanodroplet liquid phase epitaxy offered a novel approach to the formation of wide-bandgap semiconductor nanodots with size and density parameters that can be controlled by varying the absorption/desorption/nitridation times while monitor-



Authors (from left) Gozde Ozaydin, Ahmet Ozcan, and Yiyi Wang ing the evolving morphology with real-time GISAXS. A typical post-facto AFM micrograph is shown in **Figure 3**; in this case, the average GaN dot diameter is approximately 30 nm and the typical height of the dots is 2-3 nm. Separate in-situ x-ray grazing-incidence diffraction experiments show that the GaN nanodots are epitaxial and almost completely strain relaxed.

Significant variations in the adsorption/desorption behavior are observed for different substrates. Gallium desorption is significantly higher from Ga-polar GaN surfaces than from sapphire surfaces. In order to better understand the adsorption and desorption of Ga on sapphire and GaN, the surface atomic structures have to be considered. On the c-plane sapphire surface, Ga adatoms can interact directly with O terminating atoms. In contrast, on Ga-polar GaN surfaces, the Ga adatoms will interact only with other Ga atoms forming weak, delocalized Ga-Ga metallic bonds. We are preparing to investigate further how this affects the actual film growth processes.

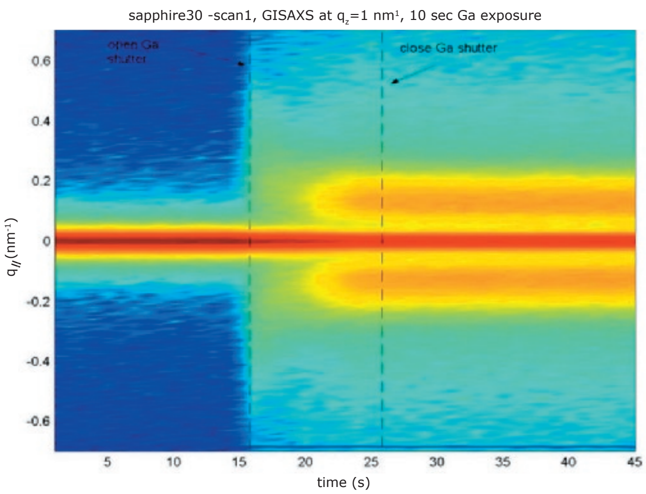


***Figure 1**. The real-time GISAXS evolution of the sapphire surface during Ga adsorption and desorption at 700 °C. The scan was taken at q_z = 1 nm^{-1}.*

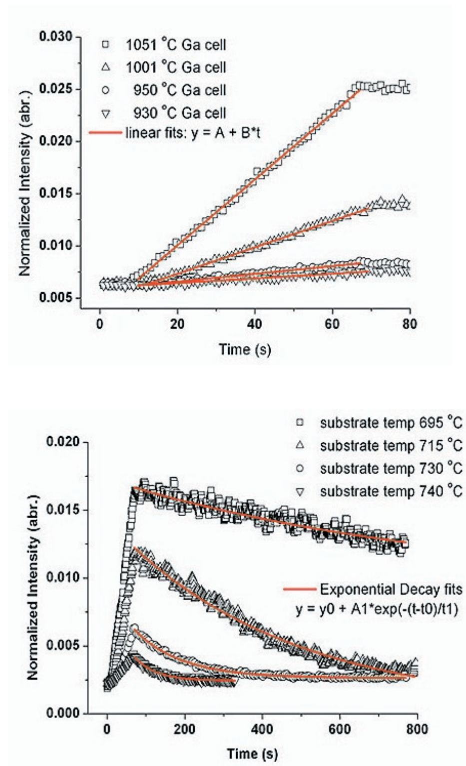


***Figure 2**. Ga fluorescence during (left) the absorption of Ga onto c-plane sapphire at different Ga cell temperatures and (right) during the desorption at different substrate temperatures.*

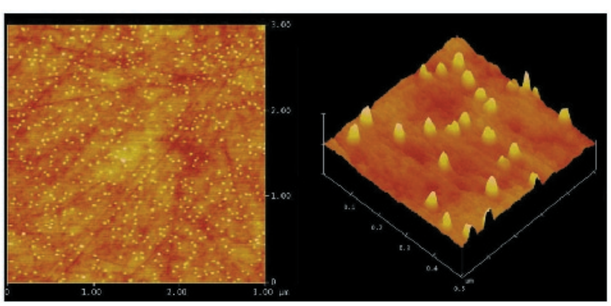


***Figure 3**. AFM analysis of the Ga nanodots on sapphire, showing a 3 x 3 micron AFM topograph. There is little or no wetting layer beneath the dots   the polishing scratch marks are still clearly visible on the sapphire substrate.*